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## Structure Reports

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***trans*-Difluoridotetrakis(pyridine- $\kappa$ N)-chromium(III) perchlorate from synchrotron radiation**Dohyun Moon<sup>a</sup> and Jong-Ha Choi<sup>b\*</sup><sup>a</sup>Pohang Accelerator Laboratory, POSTECH, Pohang 790-784, Republic of Korea, and <sup>b</sup>Department of Chemistry, Andong National University, Andong 760-749, Republic of Korea

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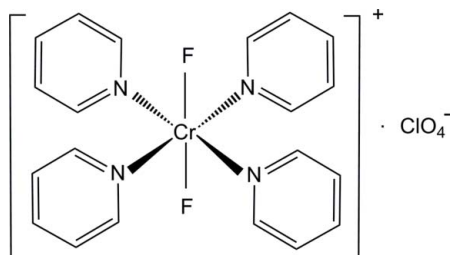
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Key indicators: single-crystal synchrotron study;  $T = 99$  K; mean  $\sigma(\text{C}—\text{C}) = 0.003$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.099; data-to-parameter ratio = 19.9.

The are two independent complex cations in the title salt,  $[\text{CrF}_2(\text{C}_5\text{H}_5\text{N})_4]\text{ClO}_4$ , each located on a centre of inversion, as well as an independent perchlorate counter-ion. The complex cations adopt slightly distorted octahedral coordination environments around the  $\text{Cr}^{\text{III}}$  ion, defined by four pyridine (py) N atoms in the equatorial plane and two  $\text{F}^-$  ligands in the axial positions; intramolecular  $\text{C}—\text{H} \cdots \text{F}$  contacts are noted. The mean  $\text{Cr}—\text{N}(\text{py})$  and  $\text{Cr}—\text{F}$  bond lengths are 2.088 (6) and 1.8559 (10) Å, respectively. The three-dimensional architecture is sustained by hydrogen bonds involving the pyridine  $\text{C}—\text{H}$  groups as donors, and F and O atoms as acceptors.

## Related literature

For background to geometric isomerism in transition metal complexes, see: Knight & Scott (2003); Ronconi & Sadler (2007). For the synthesis, see: Glerup *et al.* (1970). For the structure of *trans*- $[\text{Cr}(\text{py})_4\text{F}_2]\text{PF}_6$ , see: Fochi *et al.* (1991).



## Experimental

## Crystal data

$[\text{CrF}_2(\text{C}_5\text{H}_5\text{N})_4]\text{ClO}_4$   
 $M_r = 505.85$   
 Triclinic,  $P\bar{1}$   
 $a = 9.5690$  (19) Å  
 $b = 10.615$  (2) Å  
 $c = 12.663$  (3) Å

$\alpha = 68.46$  (3)°  
 $\beta = 68.31$  (3)°  
 $\gamma = 79.38$  (3)°  
 $V = 1109.9$  (5) Å<sup>3</sup>  
 $Z = 2$   
 Synchrotron radiation

$\lambda = 0.63001$  Å  
 $\mu = 0.49$  mm<sup>-1</sup>

$T = 99$  K  
 $0.03 \times 0.03 \times 0.02$  mm

## Data collection

ADSC Q210 CCD area-detector  
 Absorption correction: empirical  
 (*HKL3000sm*; Otwinowski & Minor, 1997)  
 $T_{\text{min}} = 0.985$ ,  $T_{\text{max}} = 0.993$

11523 measured reflections  
 5842 independent reflections  
 4912 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.099$   
 $S = 1.05$   
 5842 reflections

293 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.51$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.61$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D—H \cdots A$	$D—H$	$H \cdots A$	$D \cdots A$	$D—H \cdots A$
$\text{C1}—\text{H1} \cdots \text{F1}$	0.95	2.36	2.892 (2)	115
$\text{C6}—\text{H6} \cdots \text{F1}$	0.95	2.31	2.874 (2)	118
$\text{C11}—\text{H11} \cdots \text{F2}$	0.95	2.32	2.879 (2)	117
$\text{C16}—\text{H16} \cdots \text{F2}$	0.95	2.39	2.915 (2)	115
$\text{C14}—\text{H14} \cdots \text{O1P}$	0.95	2.61	3.154 (2)	117
$\text{C9}—\text{H9} \cdots \text{O2P}$	0.95	2.63	3.320 (3)	130
$\text{C1}—\text{H1} \cdots \text{O3P}^{\text{ii}}$	0.95	2.41	3.107 (2)	130
$\text{C4}—\text{H4} \cdots \text{O1P}^{\text{ii}}$	0.95	2.51	3.368 (2)	150
$\text{C5}—\text{H5} \cdots \text{F1}^{\text{iii}}$	0.95	2.38	2.900 (2)	114
$\text{C10}—\text{H10} \cdots \text{F1}^{\text{iii}}$	0.95	2.31	2.863 (2)	117
$\text{C15}—\text{H15} \cdots \text{F2}^{\text{iv}}$	0.95	2.29	2.856 (2)	117
$\text{C20}—\text{H20} \cdots \text{F2}^{\text{iv}}$	0.95	2.34	2.860 (2)	114
$\text{C15}—\text{H15} \cdots \text{O3P}^{\text{v}}$	0.95	2.64	3.466 (2)	146
$\text{C19}—\text{H19} \cdots \text{O2P}^{\text{vi}}$	0.95	2.58	3.227 (3)	125

Symmetry codes: (i)  $x - 1, y + 1, z$ ; (ii)  $x - 1, y, z$ ; (iii)  $-x, -y + 2, -z$ ; (iv)  $-x, -y + 2, -z + 1$ ; (v)  $-x + 1, -y + 1, -z + 1$ ; (vi)  $x, y + 1, z$ .

Data collection: *PAL ADSC Quantum-210 ADX Software* (Arvai & Nielsen, 1983); cell refinement: *HKL3000sm* (Otwinowski & Minor, 1997); data reduction: *HKL3000sm*; program(s) used to solve structure: *SHELX-2013-XS* (Sheldrick, 2008); program(s) used to refine structure: *SHELX-2013-XL* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2007); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5248).

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## supplementary materials

*Acta Cryst.* (2013). E69, m514 [doi:10.1107/S1600536813023052]

## ***trans*-Difluoridotetrakis(pyridine- $\kappa$ N)chromium(III) perchlorate from synchrotron radiation**

**Dohyun Moon and Jong-Ha Choi**

### **1. Comment**

The study of geometrical isomerism in octahedral transition metal complexes with mixed ligands has generated considerable interest, and has provided much basic structural information and spectroscopic properties (Knight & Scott, 2003). The geometry of various ligands in the metal complexes are very important in medical applications, and is likely to be a major factor in determining the anti-viral activity and its side-effects (Ronconi & Sadler, 2007). The  $[\text{Cr}(\text{py})_4\text{X}_2]^{n+}$  cation ( $\text{X}$  = monodentate;  $\text{py}$  = pyridine) can be either *trans* or *cis* geometric isomers. The infrared, electronic absorption and emission spectroscopic properties are useful in distinguishing the geometric isomers of chromium(III) complexes. However, it should be noted that the geometric and conformational assignments based on spectroscopic properties are not always definitive.

In this communication, we describe the structure of *trans*- $[\text{Cr}(\text{py})_4\text{F}_2]\text{ClO}_4$  in order to confirm the coordination of four pyridine molecules in equatorial plane and two fluoride ligands in axial positions. Counter anionic species play a very important role in coordination chemistry. This is another example of a *trans*- $[\text{Cr}(\text{py})_4\text{F}_2]^+$  structure but with a different counter anion.

The structural analysis shows the  $\text{Cr}^{\text{III}}$  complex cation to be coordinated by four nitrogen atoms of four  $\text{py}$  ligands in the equatorial sites and the two mutually *trans* fluoride atoms. The  $\text{Cr1}$  and  $\text{Cr2}$  complex cations are in half occupancy in the asymmetric unit. That is, each molecule is contributing a charge of +0.5. Thus, the salt comprises *trans*- $[\text{Cr}(\text{py})_4\text{F}_2]^+$  and  $\text{ClO}_4^-$ . An ellipsoid plot of one independent complex cation and the anion is depicted in Fig. 1.

Atoms  $\text{Cr1}$  and  $\text{Cr2}$  are located at a crystallographic center of symmetry, so these  $\text{Cr}$  complex cations have molecular  $C_i$  symmetry.

The  $\text{Cr}-\text{N}(\text{py})$  distances vary from 2.0799 (17) to 2.0929 (15) Å and the  $\text{Cr}-\text{F}$  distances are in the range of 1.8552 (10) to 1.8566 (10) Å. These bond lengths are in good agreement with those observed in *trans*- $[\text{Cr}(\text{py})_4\text{F}_2]\text{PF}_6$  (Fochi *et al.*, 1991).

The  $\text{ClO}_4^-$  anion remains outside the coordination sphere. The crystal packing is stabilized by hydrogen bonding interactions between the  $\text{C}-\text{H}$  groups of the  $\text{py}$  ligand and the oxygens of the  $\text{ClO}_4^-$  anion, Table 1. As expected, the  $\text{ClO}_4^-$  counter ion has slightly distorted tetrahedral geometry due to the influence of hydrogen bonding on the  $\text{Cl}-\text{O}$  bond lengths and the  $\text{O}-\text{Cl}-\text{O}$  angles. Consideration of the crystal packing shows that intermolecular  $\text{C}-\text{H}\cdots\text{F}$  hydrogen bonds are also present, Table 1.

### **2. Experimental**

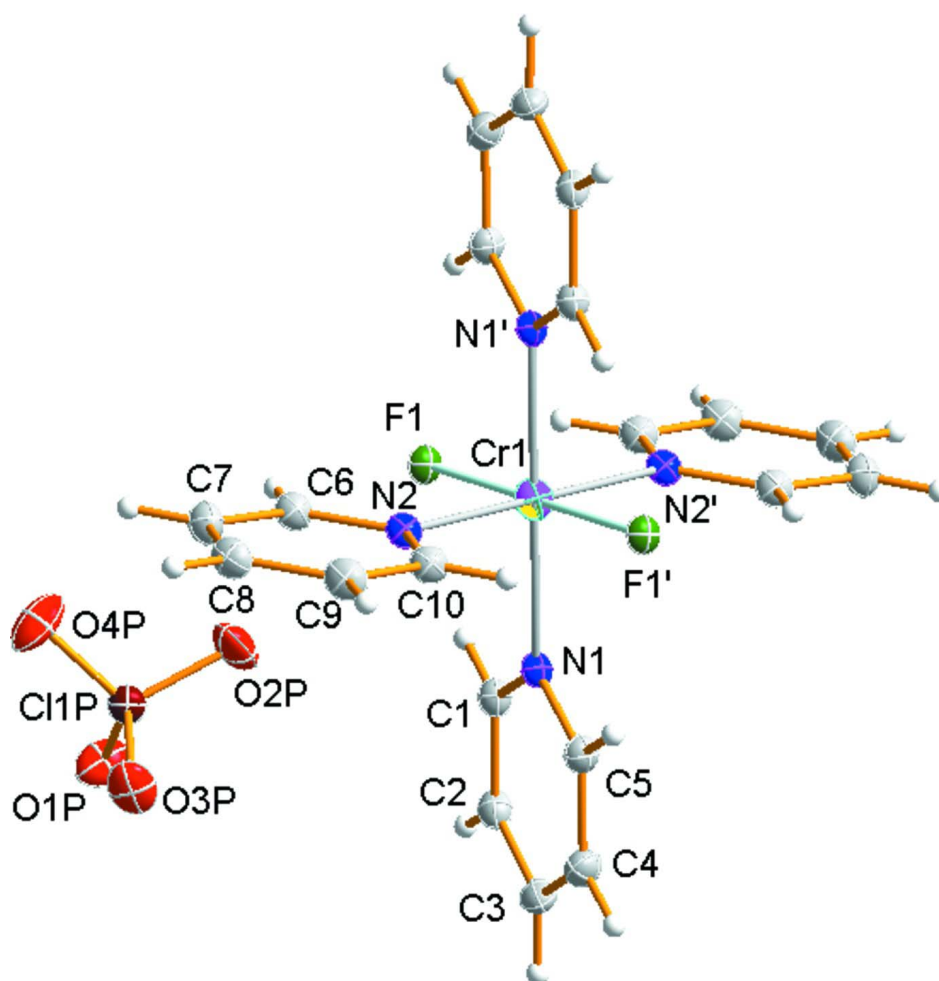
All chemicals were reagent grade materials and used without further purification. The *trans*- $[\text{Cr}(\text{py})_4\text{F}_2]\text{ClO}_4$  salt was prepared as described previously (Glerup *et al.*, 1970), and allowed to stand in 0.1 M  $\text{HClO}_4$  solution at room temperature for 1-2 days to give very small crystals suitable for X-ray structural analysis.

### 3. Refinement

C-bound H-atoms were placed in calculated positions ( $C-H = 0.95$ ) and were included in the refinement in the riding model approximation with  $U_{iso}(H)$  set to  $1.2U_{eq}(C)$ .

### Computing details

Data collection: *PAL ADSC Quantum-210 ADX Software* (Arvai & Nielsen, 1983); cell refinement: *HKL3000sm* (Otwinowski & Minor, 1997); data reduction: *HKL3000sm* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELX-2013-XS* (Sheldrick, 2008); program(s) used to refine structure: *SHELX-2013-XL* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2007); software used to prepare material for publication: *WinGX* (Farrugia, 2012).



**Figure 1**

A perspective drawing (50% probability level) of one independent complex cation and the unique perchlorate anion in the structure of *trans*-[Cr(py)<sub>4</sub>F<sub>2</sub>]ClO<sub>4</sub>

# *trans*-Difluoridotetrakis(pyridine-*κ*N)chromium(III) perchlorate

## Crystal data

[CrF<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>N)<sub>4</sub>]ClO<sub>4</sub>

*M*<sub>r</sub> = 505.85

Triclinic, *P* $\bar{1}$

Hall symbol: -*P* 1

*a* = 9.5690 (19) Å

*b* = 10.615 (2) Å

*c* = 12.663 (3) Å

$\alpha$  = 68.46 (3)°

$\beta$  = 68.31 (3)°

$\gamma$  = 79.38 (3)°

*V* = 1109.9 (5) Å<sup>3</sup>

*Z* = 2

*F*(000) = 518

*D*<sub>x</sub> = 1.514 Mg m<sup>-3</sup>

Synchrotron radiation,  $\lambda$  = 0.63001 Å

Cell parameters from 33119 reflections

$\theta$  = 0.4–33.6°

$\mu$  = 0.49 mm<sup>-1</sup>

*T* = 99 K

Pink, plate

0.03 × 0.03 × 0.02 mm

## Data collection

ADSC Q210 CCD area-detector

diffractometer

Radiation source: PLSII 2D bending magnet

$\omega$  scan

Absorption correction: empirical (using intensity measurements)

(*HKL3000sm*; Otwinowski & Minor, 1997)

*T*<sub>min</sub> = 0.985, *T*<sub>max</sub> = 0.993

11523 measured reflections

5842 independent reflections

4912 reflections with *I* > 2 $\sigma$ (*I*)

*R*<sub>int</sub> = 0.020

$\theta_{\text{max}}$  = 26.0°,  $\theta_{\text{min}}$  = 1.6°

*h* = -13→13

*k* = -14→14

*l* = -17→17

## Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R* [*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.036

*wR*(*F*<sup>2</sup>) = 0.099

*S* = 1.05

5842 reflections

293 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

*w* = 1/[ $\sigma^2(F_o^2) + (0.0543P)^2 + 0.5188P$ ]

where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3

( $\Delta/\sigma$ )<sub>max</sub> = 0.001

$\Delta\rho_{\text{max}}$  = 0.51 e Å<sup>-3</sup>

$\Delta\rho_{\text{min}}$  = -0.61 e Å<sup>-3</sup>

Extinction correction: *SHELXL*,

*F*<sub>c</sub>\* = *kF*<sub>c</sub>[1 + 0.001*xF*<sub>c</sub><sup>2</sup> $\lambda^3/\sin(2\theta)$ ]<sup>-1/4</sup>

Extinction coefficient: 0.062 (4)

## Special details

**Experimental.** Since the Pohang Accelerator Laboratory goniostat has only one omega-axis, diffn\_measured\_fraction\_theta\_full is not fully covered as 0.944, especially for the low symmetry such as a triclinic system. As this is an inherent problem, other command and option (such as *OMIT*) were not helpful to improve the completeness.

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub> */ <i>U</i> <sub>eq</sub>
Cr1	0.0000	1.0000	0.0000	0.01263 (9)
F1	0.01103 (10)	1.14193 (9)	0.04859 (8)	0.01655 (19)
N1	-0.16449 (15)	0.91455 (13)	0.16450 (12)	0.0144 (2)
C1	-0.24067 (17)	0.99130 (16)	0.23459 (14)	0.0160 (3)

H1	−0.2177	1.0836	0.2077	0.019*
C2	−0.35141 (18)	0.94021 (17)	0.34453 (15)	0.0184 (3)
H2	−0.4036	0.9969	0.3918	0.022*
C3	−0.38521 (19)	0.80573 (18)	0.38478 (15)	0.0206 (3)
H3	−0.4594	0.7682	0.4605	0.025*
C4	−0.30797 (19)	0.72648 (17)	0.31164 (16)	0.0213 (3)
H4	−0.3294	0.6341	0.3366	0.026*
C5	−0.19964 (18)	0.78444 (16)	0.20218 (15)	0.0177 (3)
H5	−0.1484	0.7307	0.1520	0.021*
N2	0.17550 (15)	0.89172 (13)	0.06286 (12)	0.0150 (3)
C6	0.24772 (18)	0.94873 (16)	0.10540 (14)	0.0182 (3)
H6	0.2131	1.0362	0.1120	0.022*
C7	0.37130 (19)	0.88404 (18)	0.14009 (16)	0.0212 (3)
H7	0.4210	0.9271	0.1691	0.025*
C8	0.42122 (19)	0.75607 (18)	0.13180 (15)	0.0214 (3)
H8	0.5052	0.7097	0.1556	0.026*
C9	0.34678 (19)	0.69668 (17)	0.08827 (16)	0.0210 (3)
H9	0.3789	0.6089	0.0817	0.025*
C10	0.22513 (18)	0.76705 (16)	0.05457 (15)	0.0181 (3)
H10	0.1746	0.7262	0.0245	0.022*
Cr2	0.0000	1.0000	0.5000	0.01280 (9)
F2	−0.15818 (10)	1.01373 (10)	0.44431 (8)	0.01706 (19)
N3	0.08874 (14)	0.82597 (13)	0.45187 (11)	0.0148 (3)
C11	0.02042 (18)	0.77691 (17)	0.40054 (15)	0.0179 (3)
H11	−0.0646	0.8267	0.3805	0.022*
C12	0.06960 (19)	0.65643 (17)	0.37590 (15)	0.0204 (3)
H12	0.0185	0.6242	0.3400	0.024*
C13	0.19391 (19)	0.58355 (17)	0.40415 (15)	0.0209 (3)
H13	0.2300	0.5010	0.3875	0.025*
C14	0.26469 (19)	0.63353 (17)	0.45721 (15)	0.0205 (3)
H14	0.3502	0.5855	0.4776	0.025*
C15	0.20934 (18)	0.75428 (17)	0.48022 (14)	0.0179 (3)
H15	0.2578	0.7877	0.5171	0.022*
N4	0.12228 (15)	1.12831 (14)	0.33344 (12)	0.0157 (3)
C16	0.05298 (18)	1.21171 (16)	0.25660 (14)	0.0177 (3)
H16	−0.0518	1.2052	0.2758	0.021*
C17	0.1294 (2)	1.30722 (17)	0.14993 (15)	0.0215 (3)
H17	0.0773	1.3660	0.0976	0.026*
C18	0.2822 (2)	1.31577 (18)	0.12077 (16)	0.0246 (4)
H18	0.3368	1.3805	0.0483	0.030*
C19	0.3546 (2)	1.2278 (2)	0.19953 (17)	0.0263 (4)
H19	0.4600	1.2304	0.1809	0.032*
C20	0.27139 (18)	1.13681 (18)	0.30494 (15)	0.0211 (3)
H20	0.3208	1.0781	0.3593	0.025*
Cl1P	0.64390 (4)	0.40483 (4)	0.24420 (3)	0.01718 (10)
O1P	0.53210 (15)	0.45637 (14)	0.33397 (13)	0.0305 (3)
O2P	0.5964 (2)	0.44001 (15)	0.14139 (13)	0.0374 (4)
O3P	0.66002 (17)	0.26002 (13)	0.29401 (13)	0.0304 (3)
O4P	0.78435 (15)	0.46338 (17)	0.20959 (16)	0.0387 (4)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cr1	0.01311 (16)	0.01227 (16)	0.01356 (16)	0.00203 (12)	−0.00396 (13)	−0.00714 (12)
F1	0.0183 (4)	0.0153 (4)	0.0190 (4)	0.0016 (3)	−0.0062 (4)	−0.0100 (3)
N1	0.0146 (6)	0.0150 (6)	0.0150 (6)	0.0008 (5)	−0.0050 (5)	−0.0071 (5)
C1	0.0153 (7)	0.0167 (7)	0.0178 (7)	0.0020 (5)	−0.0060 (6)	−0.0086 (6)
C2	0.0142 (7)	0.0245 (8)	0.0184 (7)	0.0011 (6)	−0.0046 (6)	−0.0110 (6)
C3	0.0161 (7)	0.0263 (8)	0.0183 (7)	−0.0041 (6)	−0.0036 (6)	−0.0067 (6)
C4	0.0225 (8)	0.0195 (8)	0.0223 (8)	−0.0046 (6)	−0.0066 (7)	−0.0063 (6)
C5	0.0197 (7)	0.0157 (7)	0.0195 (7)	0.0000 (6)	−0.0065 (6)	−0.0081 (6)
N2	0.0149 (6)	0.0152 (6)	0.0149 (6)	0.0021 (5)	−0.0042 (5)	−0.0069 (5)
C6	0.0188 (7)	0.0180 (7)	0.0182 (7)	0.0018 (6)	−0.0054 (6)	−0.0085 (6)
C7	0.0196 (7)	0.0241 (8)	0.0227 (8)	0.0014 (6)	−0.0089 (7)	−0.0101 (6)
C8	0.0176 (7)	0.0234 (8)	0.0212 (8)	0.0047 (6)	−0.0068 (6)	−0.0075 (6)
C9	0.0198 (7)	0.0193 (7)	0.0230 (8)	0.0061 (6)	−0.0067 (7)	−0.0098 (6)
C10	0.0185 (7)	0.0170 (7)	0.0188 (7)	0.0022 (6)	−0.0053 (6)	−0.0083 (6)
Cr2	0.00999 (15)	0.01573 (17)	0.01295 (16)	0.00214 (12)	−0.00418 (12)	−0.00595 (12)
F2	0.0138 (4)	0.0204 (5)	0.0191 (4)	0.0021 (3)	−0.0077 (4)	−0.0080 (4)
N3	0.0133 (6)	0.0163 (6)	0.0135 (6)	0.0011 (5)	−0.0033 (5)	−0.0057 (5)
C11	0.0153 (7)	0.0199 (7)	0.0187 (7)	0.0013 (6)	−0.0056 (6)	−0.0076 (6)
C12	0.0190 (7)	0.0213 (8)	0.0225 (8)	0.0003 (6)	−0.0065 (6)	−0.0101 (6)
C13	0.0200 (7)	0.0180 (7)	0.0218 (8)	0.0023 (6)	−0.0035 (6)	−0.0084 (6)
C14	0.0172 (7)	0.0214 (8)	0.0198 (7)	0.0056 (6)	−0.0053 (6)	−0.0072 (6)
C15	0.0146 (7)	0.0220 (8)	0.0176 (7)	0.0033 (6)	−0.0055 (6)	−0.0087 (6)
N4	0.0140 (6)	0.0183 (6)	0.0149 (6)	0.0007 (5)	−0.0035 (5)	−0.0076 (5)
C16	0.0175 (7)	0.0193 (7)	0.0165 (7)	0.0023 (6)	−0.0056 (6)	−0.0077 (6)
C17	0.0266 (8)	0.0191 (7)	0.0178 (7)	0.0018 (6)	−0.0068 (7)	−0.0068 (6)
C18	0.0272 (9)	0.0224 (8)	0.0196 (8)	−0.0054 (7)	−0.0003 (7)	−0.0073 (6)
C19	0.0183 (8)	0.0327 (10)	0.0242 (8)	−0.0051 (7)	−0.0013 (7)	−0.0090 (7)
C20	0.0149 (7)	0.0275 (8)	0.0194 (7)	−0.0010 (6)	−0.0046 (6)	−0.0073 (6)
Cl1P	0.01680 (17)	0.01445 (17)	0.02051 (18)	0.00369 (12)	−0.00615 (14)	−0.00815 (13)
O1P	0.0234 (6)	0.0276 (7)	0.0369 (8)	0.0019 (5)	0.0019 (6)	−0.0201 (6)
O2P	0.0580 (10)	0.0308 (8)	0.0269 (7)	−0.0049 (7)	−0.0253 (7)	−0.0007 (6)
O3P	0.0427 (8)	0.0139 (6)	0.0382 (8)	0.0093 (5)	−0.0219 (7)	−0.0092 (5)
O4P	0.0178 (6)	0.0411 (8)	0.0597 (10)	−0.0049 (6)	−0.0017 (7)	−0.0291 (8)

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

Cr1—F1 <sup>i</sup>	1.8566 (10)	Cr2—N4	2.0799 (17)
Cr1—F1	1.8566 (10)	Cr2—N4 <sup>ii</sup>	2.0800 (17)
Cr1—N1 <sup>i</sup>	2.0867 (16)	Cr2—N3 <sup>ii</sup>	2.0908 (14)
Cr1—N1	2.0867 (16)	Cr2—N3	2.0908 (14)
Cr1—N2 <sup>i</sup>	2.0929 (15)	N3—C11	1.345 (2)
Cr1—N2	2.0929 (15)	N3—C15	1.350 (2)
N1—C1	1.3478 (19)	C11—C12	1.385 (2)
N1—C5	1.348 (2)	C11—H11	0.9500
C1—C2	1.386 (2)	C12—C13	1.383 (2)
C1—H1	0.9500	C12—H12	0.9500
C2—C3	1.384 (2)	C13—C14	1.386 (3)

C2—H2	0.9500	C13—H13	0.9500
C3—C4	1.396 (2)	C14—C15	1.385 (2)
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.386 (2)	C15—H15	0.9500
C4—H4	0.9500	N4—C16	1.341 (2)
C5—H5	0.9500	N4—C20	1.348 (2)
N2—C6	1.343 (2)	C16—C17	1.387 (2)
N2—C10	1.350 (2)	C16—H16	0.9500
C6—C7	1.388 (2)	C17—C18	1.382 (3)
C6—H6	0.9500	C17—H17	0.9500
C7—C8	1.383 (2)	C18—C19	1.391 (3)
C7—H7	0.9500	C18—H18	0.9500
C8—C9	1.386 (3)	C19—C20	1.378 (3)
C8—H8	0.9500	C19—H19	0.9500
C9—C10	1.381 (2)	C20—H20	0.9500
C9—H9	0.9500	Cl1P—O4P	1.4342 (15)
C10—H10	0.9500	Cl1P—O3P	1.4342 (14)
Cr2—F2 <sup>ii</sup>	1.8552 (10)	Cl1P—O2P	1.4351 (15)
Cr2—F2	1.8552 (10)	Cl1P—O1P	1.4416 (14)
F1 <sup>i</sup> —Cr1—F1	180.0	F2 <sup>ii</sup> —Cr2—N4 <sup>ii</sup>	90.36 (6)
F1 <sup>i</sup> —Cr1—N1 <sup>i</sup>	90.52 (5)	F2—Cr2—N4 <sup>ii</sup>	89.64 (6)
F1—Cr1—N1 <sup>i</sup>	89.48 (5)	N4—Cr2—N4 <sup>ii</sup>	180.0
F1 <sup>i</sup> —Cr1—N1	89.48 (5)	F2 <sup>ii</sup> —Cr2—N3 <sup>ii</sup>	90.31 (5)
F1—Cr1—N1	90.52 (5)	F2—Cr2—N3 <sup>ii</sup>	89.69 (5)
N1 <sup>i</sup> —Cr1—N1	180.00 (7)	N4—Cr2—N3 <sup>ii</sup>	87.08 (6)
F1 <sup>i</sup> —Cr1—N2 <sup>i</sup>	90.40 (5)	N4 <sup>ii</sup> —Cr2—N3 <sup>ii</sup>	92.92 (6)
F1—Cr1—N2 <sup>i</sup>	89.60 (5)	F2 <sup>ii</sup> —Cr2—N3	89.68 (5)
N1 <sup>i</sup> —Cr1—N2 <sup>i</sup>	92.68 (6)	F2—Cr2—N3	90.32 (5)
N1—Cr1—N2 <sup>i</sup>	87.32 (6)	N4—Cr2—N3	92.92 (6)
F1 <sup>i</sup> —Cr1—N2	89.60 (5)	N4 <sup>ii</sup> —Cr2—N3	87.08 (6)
F1—Cr1—N2	90.40 (5)	N3 <sup>ii</sup> —Cr2—N3	180.0
N1 <sup>i</sup> —Cr1—N2	87.32 (6)	C11—N3—C15	118.33 (14)
N1—Cr1—N2	92.68 (6)	C11—N3—Cr2	120.91 (11)
N2 <sup>i</sup> —Cr1—N2	180.0	C15—N3—Cr2	120.55 (11)
C1—N1—C5	118.53 (14)	N3—C11—C12	122.38 (15)
C1—N1—Cr1	119.68 (11)	N3—C11—H11	118.8
C5—N1—Cr1	121.77 (11)	C12—C11—H11	118.8
N1—C1—C2	122.25 (15)	C13—C12—C11	119.25 (16)
N1—C1—H1	118.9	C13—C12—H12	120.4
C2—C1—H1	118.9	C11—C12—H12	120.4
C3—C2—C1	119.33 (15)	C12—C13—C14	118.63 (15)
C3—C2—H2	120.3	C12—C13—H13	120.7
C1—C2—H2	120.3	C14—C13—H13	120.7
C2—C3—C4	118.55 (15)	C15—C14—C13	119.30 (15)
C2—C3—H3	120.7	C15—C14—H14	120.4
C4—C3—H3	120.7	C13—C14—H14	120.4
C5—C4—C3	119.09 (16)	N3—C15—C14	122.11 (15)
C5—C4—H4	120.5	N3—C15—H15	118.9

C3—C4—H4	120.5	C14—C15—H15	118.9
N1—C5—C4	122.22 (15)	C16—N4—C20	118.68 (15)
N1—C5—H5	118.9	C16—N4—Cr2	120.86 (11)
C4—C5—H5	118.9	C20—N4—Cr2	120.25 (12)
C6—N2—C10	118.41 (14)	N4—C16—C17	122.13 (16)
C6—N2—Cr1	120.40 (10)	N4—C16—H16	118.9
C10—N2—Cr1	121.05 (11)	C17—C16—H16	118.9
N2—C6—C7	122.17 (15)	C18—C17—C16	119.13 (17)
N2—C6—H6	118.9	C18—C17—H17	120.4
C7—C6—H6	118.9	C16—C17—H17	120.4
C8—C7—C6	119.12 (16)	C17—C18—C19	118.76 (17)
C8—C7—H7	120.4	C17—C18—H18	120.6
C6—C7—H7	120.4	C19—C18—H18	120.6
C7—C8—C9	118.91 (16)	C20—C19—C18	119.08 (17)
C7—C8—H8	120.5	C20—C19—H19	120.5
C9—C8—H8	120.5	C18—C19—H19	120.5
C10—C9—C8	119.00 (15)	N4—C20—C19	122.20 (17)
C10—C9—H9	120.5	N4—C20—H20	118.9
C8—C9—H9	120.5	C19—C20—H20	118.9
N2—C10—C9	122.39 (16)	O4P—Cl1P—O3P	110.13 (10)
N2—C10—H10	118.8	O4P—Cl1P—O2P	109.84 (11)
C9—C10—H10	118.8	O3P—Cl1P—O2P	109.44 (9)
F2 <sup>ii</sup> —Cr2—F2	180.0	O4P—Cl1P—O1P	109.02 (9)
F2 <sup>ii</sup> —Cr2—N4	89.64 (6)	O3P—Cl1P—O1P	108.90 (9)
F2—Cr2—N4	90.36 (6)	O2P—Cl1P—O1P	109.50 (10)
C5—N1—C1—C2	1.2 (2)	C15—N3—C11—C12	−0.1 (2)
Cr1—N1—C1—C2	179.81 (12)	Cr2—N3—C11—C12	−175.00 (12)
N1—C1—C2—C3	0.3 (2)	N3—C11—C12—C13	−0.4 (3)
C1—C2—C3—C4	−1.2 (2)	C11—C12—C13—C14	0.4 (3)
C2—C3—C4—C5	0.5 (3)	C12—C13—C14—C15	0.0 (2)
C1—N1—C5—C4	−1.9 (2)	C11—N3—C15—C14	0.5 (2)
Cr1—N1—C5—C4	179.55 (13)	Cr2—N3—C15—C14	175.44 (12)
C3—C4—C5—N1	1.0 (3)	C13—C14—C15—N3	−0.5 (2)
C10—N2—C6—C7	0.4 (2)	C20—N4—C16—C17	−0.9 (2)
Cr1—N2—C6—C7	−175.43 (12)	Cr2—N4—C16—C17	173.85 (12)
N2—C6—C7—C8	−0.6 (3)	N4—C16—C17—C18	0.9 (2)
C6—C7—C8—C9	0.4 (3)	C16—C17—C18—C19	0.1 (2)
C7—C8—C9—C10	0.0 (3)	C17—C18—C19—C20	−1.2 (3)
C6—N2—C10—C9	0.1 (2)	C16—N4—C20—C19	−0.3 (2)
Cr1—N2—C10—C9	175.84 (12)	Cr2—N4—C20—C19	−175.00 (14)
C8—C9—C10—N2	−0.2 (3)	C18—C19—C20—N4	1.3 (3)

Symmetry codes: (i)  $-x, -y+2, -z$ ; (ii)  $-x, -y+2, -z+1$ .

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1 $\cdots$ F1	0.95	2.36	2.892 (2)	115
C6—H6 $\cdots$ F1	0.95	2.31	2.874 (2)	118

C11—H11...F2	0.95	2.32	2.879 (2)	117
C16—H16...F2	0.95	2.39	2.915 (2)	115
C14—H14...O1 <sup>P</sup>	0.95	2.61	3.154 (2)	117
C9—H9...O2 <sup>P</sup>	0.95	2.63	3.320 (3)	130
C1—H1...O3 <sup>P</sup> <sup>iii</sup>	0.95	2.41	3.107 (2)	130
C4—H4...O1 <sup>P</sup> <sup>iv</sup>	0.95	2.51	3.368 (2)	150
C5—H5...F1 <sup>i</sup>	0.95	2.38	2.900 (2)	114
C10—H10...F1 <sup>i</sup>	0.95	2.31	2.863 (2)	117
C15—H15...F2 <sup>ii</sup>	0.95	2.29	2.856 (2)	117
C20—H20...F2 <sup>ii</sup>	0.95	2.34	2.860 (2)	114
C15—H15...O3 <sup>P</sup> <sup>v</sup>	0.95	2.64	3.466 (2)	146
C19—H19...O2 <sup>P</sup> <sup>vi</sup>	0.95	2.58	3.227 (3)	125

Symmetry codes: (i)  $-x, -y+2, -z$ ; (ii)  $-x, -y+2, -z+1$ ; (iii)  $x-1, y+1, z$ ; (iv)  $x-1, y, z$ ; (v)  $-x+1, -y+1, -z+1$ ; (vi)  $x, y+1, z$ .